

22005年12月20日19時53分

JRICOH-RTR SYH3MD

NNO. 0923 PP. 2

DEC. 13. 2005 5:09PM

OBLON SPIVAK

NO. 075 P. 16

Docket No.: 252035US0DIV

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

IN RE APPLICATION OF:

GROUP: 1714

Nozomu TAMOTO, et al.

SERIAL NO: 10/827,376

EXAMINER: VICKEY RONESI

FILED: April 20, 2004

FOR: ELECTROPHOTOGRAPHIC PHOTORECEPTOR, AND IMAGE FORMING  
METHOD AND APPARATUS USING THE PHOTORECEPTOR

**DECLARATION UNDER 37 C.F.R. 1.132**

COMMISSIONER FOR PATENTS  
ALEXANDRIA, VIRGINIA 22313

Sir:

Now comes Nozomu Tamoto who deposes and states that

1. I am a graduate of Nagasaki University  
of Technology and received my master degree in the year

1992

2. I have been employed by Ricoh co. Ltd. for 13 years as an engineer  
in the field of electro photography

3. The following experiments were carried out by me or under my direct supervision  
and control.

Experimental Data

1. Effects of ball milling

The following components were mixed and dispersed under the below-mentioned  
conditions.

Alumina

13 g

(SUMICORUNDUM AA-03 from Sumitomo Chemical Co., Ltd., average primary

particle diameter of about 0.3  $\mu\text{m}$ , resistivity of not less than  $10^{10} \Omega \text{ cm}$ , and pH of from 8 to 9)

Wetting dispersant

0.52 g

(BYK-P104 from BYK Chemie, solution of unsaturated polycarboxylic acid polymer,  
acid value of about 180

mgKOH/g, and solid content of 50 %)

Cyclohexanone

318 g

(in a case of solid content of 4%)

14.1 g

(in a case of solid content of 48 %)

119 g

(in a case of solid content of 10 %)

The dispersing conditions were as follows.

Dispersion condition	Dispersing device	Solid content of dispersion	Dispersing time
A	Ball mill	48 %	24 hours
B	Ball mill	4 %	24 hours
C	Stirrer	4 %	6 days
D	Bead mill	10 %	20 minutes
F	Bead mill	10 %	1 hour
F	Ultrasonic dispersing machine	4 %	30 minutes
G	Ultrasonic dispersing machine	4 %	2 hours
H	Ultrasonic dispersing machine	4 %	6 hours

#### Evaluation method

Each of the dispersions prepared above was contained in a test tube and was allowed to settle up to 100 hours while the height (H<sub>0</sub>) of the clouded portion, which is illustrated in FIG. 11, was measured at regular intervals. The results are shown in FIG. 12. FIGs. 11 and 12 are attached herewith and incorporated by reference into the Declaration.

It is clear from FIG. 12 that the height of the clouded portion of the dispersions prepared with a ball mill is longer than those of the other dispersions prepared with other

dispersing devices such as a stirrer, a bead mill and an ultrasonic dispersing machine. Namely, the precipitating speed of the filler in the dispersions prepared by ball milling is slower than those in the cases using the other dispersions, i.e., the dispersions prepared with ball milling have good dispersion stability.

The present invention is that an organic compound having a specific acid value is adsorbed to the surface of a filler, and thereby the wettability of the filler is enhanced while contact among the filler particles is limited, resulting in prevention of aggregation of the filler particles.

By using this technique of the present invention, a filler can be finely dispersed while the dispersion has good dispersion stability. In contrast, when a filler is dispersed (i.e., pulverized) with a bead mill or an ultraviolet dispersing machine, which can apply a strong force to the filler, the pulverized filler particles have an active surface. However, it is considered that an organic compound having a specific acid value cannot be fully adsorbed to the active surface. When a filler is dispersed with a stirrer, the filler is hardly pulverized (i.e., the aggregates of the filler are hardly dissociated). Therefore, an organic compound cannot be fully adsorbed to the surface of each particle of the filler, and thereby the dispersibility of the dispersion cannot be improved.

In contrast, when a filler is dispersed with a ball mill, the filler particles are gradually pulverized (like dissociating aggregates of the filler particles) because a relatively weak force is applied thereto. In addition, all the filler particles are almost uniformly pulverized and dispersed in the dispersion, and thereby an organic compound is securely adsorbed to active surfaces of the pulverized filler particles, resulting in enhancement of the wettability of the filler. In addition, contact among the filler particles is limited due to steric hindrance, and thereby the dispersing stability of the dispersion can be dramatically improved.

## 2. Effects of alumina ball.

The following components were mixed.

Alumina

1.26 g

(SUMICORUNDUM AA-03 from Sumitomo Chemical Co., Ltd., average primary

particle diameter of about 0.3  $\mu\text{m}$ , resistivity of not less than  $10^{10} \Omega \text{ cm}$ , and pH of from 8 to 9)

Cyclohexanone

19.74 g

The mixture was dispersed with a ball mill while changing the diameter and material of the balls as follows.

The dispersing conditions were as follows.

Condition	Material of ball (weight of ball)	Diameter of ball	Solid content of dispersion	Dispersing time
I	Zirconia (PSZ) (96-100 g)	5 mm	6 %	2 hours
J	Zirconia (PSZ) (96-100 g)	5mm	6%	4days
K	Zirconia (PSZ) (96-100 g)	5 mm	6 %	27 days
L	Zirconia (PSZ) (96-100 g)	10 mm	6 %	4 days
M	Alumina (73 g)	5mm	6%	4days
N	Alumina (73 g)	10mm	6%	4days
O	Glass (47-50 g)	5mm	6%	4days
P	Agate (47-50 g)	10mm	6%	4days

#### Evaluation method

Each of the dispersions mentioned above was contained in a test tube and was allowed to settle up to 100 hours while the height ( $H_c$ ) of the clouded portion and the height ( $H_p$ ) of the precipitate, which are illustrated in FIG. 11, was measured. The results are shown in FIGS. 13 and 14. FIGs. 11-15 are attached herewith and incorporated by reference into the Declaration.

The following can be understood from FIGs. 13 and 14. (1) When zirconia balls (PSZ) are used for a ball mill, the filler in the resultant dispersion precipitates rapidly. Namely, the dispersion has poor dispersion stability. As the dispersing time is increased, the height ( $H_c$ ) of the clouded portion is increased but the height ( $H_p$ ) of the precipitate is also increased. Therefore, it is considered that the filler particles aggregate.

(2) When a glass ball and an agate ball are used for a ball mill, the filler in the resultant dispersion precipitates rapidly. Namely, the dispersion has poor dispersion stability.

(3) In contrast, when alumina balls are used, the height ( $H_c$ ) of the clouded portion is relatively long and the height ( $H_p$ ) of the precipitate is relatively short compared to the other cases. Namely, the dispersion has good dispersion stability.

Thus, even when a ball mill is used, the dispersibility and dispersion stability of the resultant dispersions greatly change depending on the dispersion media (i.e., balls). The reason therefor is not determined but is considered to be that the powders generated by abrasion of the dispersing media influence the dispersibility and dispersion stability of the resultant dispersion.

FIG. 15 is a graph illustrating the ratio of the weight of powders generated by abrasion of the dispersing media to the original weight of the balls, which is determined by the difference between the weights of the balls before and after the dispersing operation. It is clear from FIG. 15 that a considerable amount of ball powders are included in the resultant dispersion regardless of the balls used, and the dispersion stability of the dispersions greatly changes depending on the materials of the balls. When an alumina ball is used, the dispersion stability of the dispersion is hardly influenced by the generated ball powders, rather the dispersion stability is improved.

As a result of this experiment, it is found that by dispersing a filler with a ball mill using an alumina ball, not only the filler can be finely dispersed, but also the dispersion stability of the dispersion can be improved. This is because an organic compound is well adsorbed to the surface of the pulverized filler, and thereby the dispersibility and dispersion stability of the resultant dispersion can be improved. Since the filler is well dispersed in the

resultant photoreceptor, occurrence of the problem in that the residual potential increases can be prevented.

Namely, by preparing a coating liquid using a ball mill containing an alumina ball as recited in Claim 27, the effects of the present invention can be well produced.

4. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

5. Further deponent saith not.

Customer Number

22850

Tel. (703) 413-3000  
Fax. (703) 413-2220  
(OSMDMN 07/05)

Signature

*Nozomu Yamato*

Date

*December 20, 2005*

**RICOH**

FIG. 11

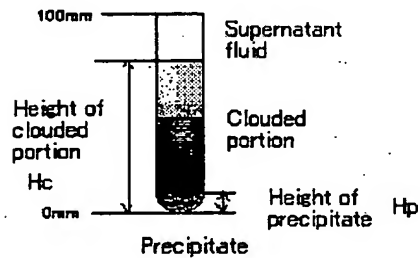
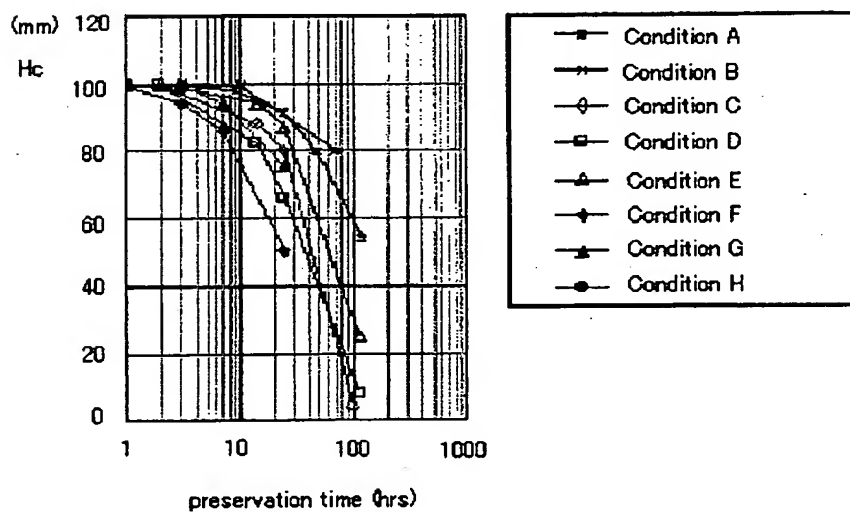


FIG. 12



**RICOH**

FIG. 13

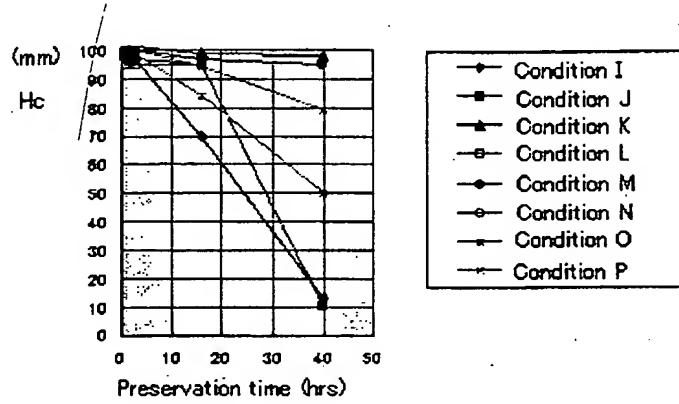
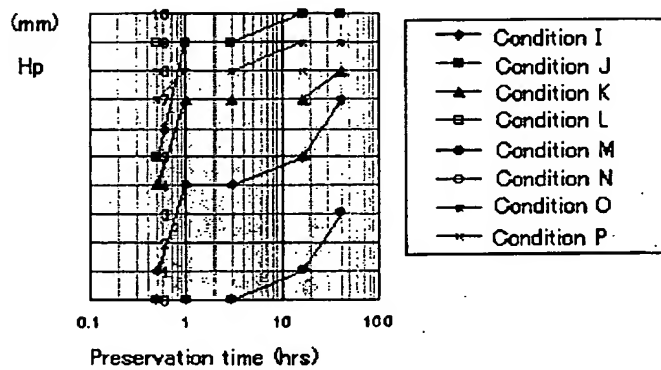


FIG. 14





**RICOH**

FIG. 15

